भारतीय मानक
Indian Standard

IS 439 : 2020

औद्योगिक कोक — विशिष्टि

(चौथा पुनरीक्षण)

Industrial Coke — Specification

(Fourth Revision)

ICS 73.040

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Fourth Revision) was adopted by Bureau of Indian Standards after the draft finalized by the Solid Mineral Fuels and Solid Biofuels Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1953 and subsequently revised in 1965, 1976 and 1989. In the second revision, the series of standards on coke for specific purposes were withdrawn and a consolidated standard on coke was formulated.

In the third revision, details of methods for determination of ash fusion temperature and reactivity of coke were excluded since they were covered in separate standards. Besides, new grades were incorporated, in the case of coke for gas making (GC) and coke for ferro-alloys industry (FAC) and foundry coke (FC) existing grades were modified, accommodating cokes with higher ash content in view of the prevailing conditions in the industry. No changes were made in the case of blast furnace coke (BFC).

The fourth revision has been taken up to address the developments in coke production plants. 'Coke for Gas Making (GC)' has been removed from scope of standard as there are no producers and users of Gas Coke in the country. In this revision, the following changes have been made in 'Requirements for Industrial Coke' (Table 1):

a) The volatile matter and phosphorous requirements of BFC have been reduced to 1 percent (*Max*) and 0.15 percent (*Max*), respectively;

b) Coke strength after reaction (CSR) and coke reactivity index (CRI) are important means of evaluating the quality of coke and of controlling BFC. Therefore, the limits of CSR and CRI have been specified for BFC; and

c) Size range requirement of FAC has been changed.

Further, the test method for determination of reactivity of coke to carbon dioxide as given in IS 4023 : 1991 has been reproduced in Annex B.

In the formulation of this standard, due weightage has been given to international coordination among the standards and practices prevailing in different countries in addition to relating it to the practice in the fields in this country. This has been met by deriving assistance from investigations carried out at the CSIR-Central Institute of Mining and Fuel Research, Dhanbad.

The composition of the Committee responsible for the formulation of this Indian Standard is given at Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

INDUSTRIAL COKE — SPECIFICATION

(Fourth Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for industrial coke suitable for use in blast furnaces, foundries and ferro-alloys industry.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
437 : 2020	Size analysis of coal and coke for marketing (*fourth revision*)
1350 (Part 1) : 1984	Methods of test for coal and coke: Part 1 Proximate analysis (*second revision*)
1350 (Part 3) : 1969	Methods of test for coal and coke: Part 3 Determination of sulfur (*first revision*)
1350 (Part 5) : 2017	Methods of test for coal and coke: Part 5 Special impurities (*first revision*)
1354 : 1992	Methods of test for coal — Special tests (*second revision*)
4023 : 1991	Methods for the determination of reactivity of coke (*first revision*)
16143 (Part 2) : 2014	Hard coal and coke-mechanical mechanical sampling: Part 2 Sampling from moving streams

3 DESIGNATION AND GRADES

3.1 Designations

Industrial coke for various uses shall be designated as follows:

Industrial Coke	*Designation*
Blast Furnace Coke	BFC
Foundary Coke	FC
Coke for Ferro-Alloys Industry	FAC

3.2 Grades

Coke shall be of the following grades based on ash content as specified in Table 1.

a) *Blast furnace coke* — Grade 1 and Grade 2.

b) *Foundary coke* — Special Grade, Grade 1, Grade 2 and Grade 3.

c) *Coke for ferro-alloys industry* — Special Grade, Grade 1, Grade 2 and Grade 3.

4 REQUIREMENTS

4.1 Coke shall conform to the requirements prescribed in Table 1 when tested in accordance with the methods as prescribed in column 13 of Table 1.

4.2 Size Ranges

Size ranges for coke for various purposes at the point of loading ex-plant shall be as specified at Sl No. (xii) of Table 1.

5 BIS CERTIFICATION MARKING

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 Representative samples of coke shall be drawn as prescribed in IS 16143 (Part 2).

NOTE — In the case of blast furnace coke, the samples shall be drawn from the skip or from a point immediately before the skip. In case it is not possible to draw it from the skip, the representative samples shall first be drawn from the wharf and then conditioned for stabilization as prescribed in Annex A.

Table 1 Requirements for Industrial Coke

(*Clauses* 3.2, 4.1 *and* 4.2)

Sl No.	Characteristic	Requirements										Method of Test, Ref to IS/Annex
	Coke Designation	Blast Furnace Coke (BFC)		Foundry Coke (FC)				Coke for Ferroalloys Industry (FAC)				
		Grade 1	Grade 2	Special Grade	Grade 1	Grade 2	Grade 3	Special Grade	Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	Ash percent, *Max*	22	25	20	25	30	35	20	22	25	30	IS 1350 (Part 1)
ii)	Moisture percent, *Max*	4	4	4	4	4	4	5	5	5	5	IS 1350 (Part 1)
iii)	Volatile matter percent, *Max*	1.0	1.5	2.0	2.0	2.0	2.0	2.0	3.0	3.0	3.0	IS 1350 (Part 1)
iv)	Sulphur percent, *Max*	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	IS 1350 (Part 3)
v)	Phosphorus percent, *Max*	0.15	0.25	0.15	0.3	0.3	0.3	0.04	0.10	0.16	0.16	**5** of IS 1350 (Part 5)
vi)	Shatter index *Min*											**4** of IS 1354
	a) Over 50 mm	—	—	90	85	80	75	—	—	—	—	
	b) Over 12.5 mm	—	—	96	95	94	92	—	—	—	—	
vii)	Micum index											**5** of IS 1354
	a) +40 mm *Min*	78	76	—	—	—	—	—	—	—	—	
	b) –10 mm *Max*	10	12	—	—	—	—	—	—	—	—	
viii)	Porosity percent	38 to 45	38 to 45	35 to 45	35 to 45	35 to 45	35 to 45	—	—	—	—	**8** of IS 1354
ix)	Reactivity to CO_2	—	—	—	—	—	—	180	150	120	120	Annex B
xii)	Coke strength after reaction (CSR)	60-62	58-60	—	—	—	—	—	—	—	—	IS 4023
xiii)	Coke Reactivity Index (CRI)	21-23	22-25	—	—	—	—	—	—	—	—	IS 4023
xiv)	Size range	The material shall be in the size range between 100 and 25 mm with a size tolerance of 5 percent by mass for oversize and 10 percent by mass for undersize.		Size of coke shall be as agreed to between the purchaser and the supplier.				The material shall be in 3 size ranges, namely between 50 and 25 mm; 25 and 5 mm and 20 and 6.3 mm.				IS 437

ANNEX A

(*Clause* 5.1, *Note*)

METHODS OF STABILIZING COKE

A-1 GENERAL

In case when the sample of coke has to be drawn from the coke wharf, as in the case of merchant cookeries, pre-treatment of the same is necessary to bring it to the approximate condition it would have attained in its movement from the wharf to the blast furnace end.

A-2 APPARATUS

The equipment used is similar to that prescribed for 'shatter test' in **4** and Fig. 1 of IS 1354.

A-3 PROCEDURE

Collect about 400 kg of coke above 50 mm in size and divide in 8 lots of 50 kg each. Fill the box with a lot of 50 kg carefully placing in it each lump by hand. Raise the box to the height of 2 meters, close and latch the front plate of the base and displace the latch of the box to allow the coke to fall on the base plate. Repeat the above procedure with the same sample. Then place the coke in lots of 50 kg each into the micum test drum and rotate the drum for one minute making 25 revolutions. Screen the coke thus treated through a 50 mm **s**quare hole sieve and reject the material passing through it. Then screen the coke retained on the 50 mm sieve further through 125, 100 and 75 mm sieves and draw samples for physical tests, from the graded coke as prescribed in IS 16143 (Part 2).

NOTE — The treatment of dropping coke sample twice from a height of 2 m is to be given to all the 8 lots of 50 kg each separately.

ANNEX B

[*Table* 1, *Sl. No.* (ix)]

DETERMINATION OF REACTIVITY OF COKE TO CARBON DIOXIDE

B-1 Two methods for determination of reactivity of coke to carbon dioxide have been prescribed, namely, Method A and Method B.

B-1.1 Method A

B-1.1.0 *Outline of the Method*

A standard reaction tube, containing graded coke sample, is heated from room temperature to the specified reaction temperature. After attaining standard conditions, 100 ml of carbon dioxide is passed at a definite rate through the coke bed and the volume of carbon monoxide formed is measured to give the reactivity value.

B-1.1.1 *Standard Conditions*

The standard conditions to be maintained for the determination of reactivity of coke shall be as follows:

a)	Temperate of coke	950 ± 2°C
b)	Rate of passage of carbon-dioxide	5 ml/min
c)	Volume of carbon dioxide to be passed for each determination	100 ml
d)	Length of column of coke in reaction tube (with thermocouple in position)	7.5 cm
e)	Size of coke	Between 0.6 and 1.2 mm
f)	Normal temperature	0°C (273 K)
g)	Normal pressure	133.322 N/m^2)

B-1.1.2 *Apparatus*

The working arrangement of the apparatus is illustrated in Fig. 1. The set-up consists of the following main constituents.

B-1.1.2.1 *Preheater furnace*

Maintained at 600 ± 2°C and containing copper for removing any oxygen in the gases and preheating prior to entering the reaction tube.

B-1.1.2.2 *Gas washers*

Two, one containing alkaline pyrogallol solution and the other concentrated sulphuric acid.

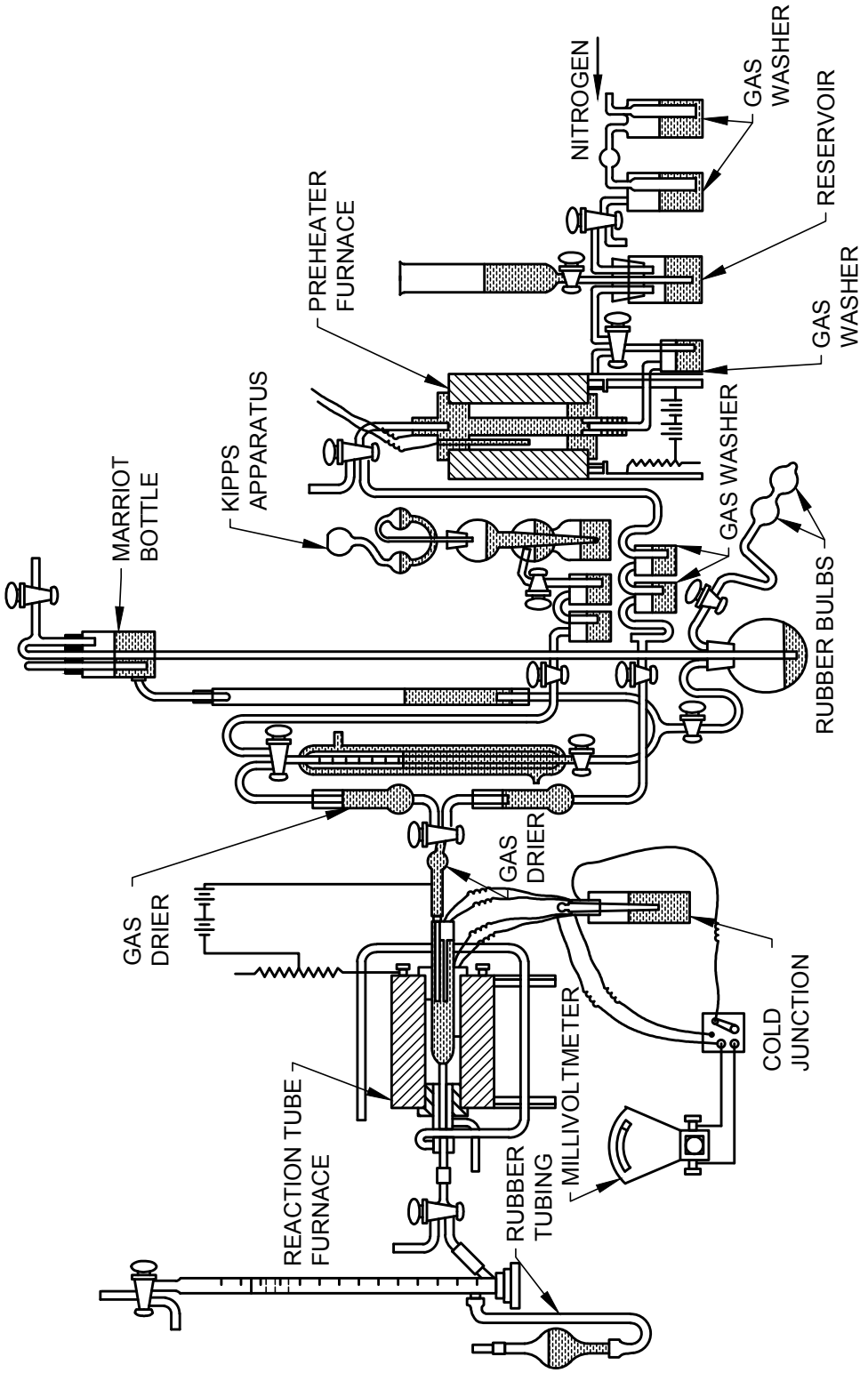


FIG. 1 APPRATUS FOR DETERMINING THE RECATIVITY OF COKE

B-1.1.2.3 *Gas driers*

Three, containing concentrated sulphuric acid, calcium chloride and phosphoric oxide respectively.

B-1.1.2.4 *Reservoir*

Containing concentrated sulphuric acid and arranged so that the whole apparatus can be left under pressure of nitrogen whilst both the furnace and reaction tube are being cooled. It also enables the pyrogallol wash solution to be changed without disturbing other parts of the apparatus.

B-1.1.2.5 *Marriot bottle*

Containing concentrated magnesium chloride solution for expelling carbon dioxide at a constant rate of flow into the side tube. This bottle is fitted with a capillary air tube and a jet so arranged that it delivers approximately 5 ml of gas per minute. The jet is connected to the bottle by means of a bent glass tube and a short length of rubber tubing. By movement of the jet in the tubing the head of liquid can be varied and the rate of flow altered.

NOTE ─ This method of variation has been found to be much easier to control than a tap placed in the system.

B-1.1.2.6 *Reaction tube furnace*

Having a tube, 30 cm in length and 5 cm in internal diameter, and wound on the outside with nichrome wire so that a uniform temperature of 950 ± 2°C might be attained at least through a length of 8 cm in the middle portion. The reaction tube is made of silica. A piston of steatite just slides into the silica tube and a longer silica capillary tube, also a sliding fit, is secured to the silica sheath by a packing of asbestos or kieselguhr. The whole assembly is fitted into the reaction tube by means of the rubber bung containing two holes one (central) for the sheath, and the other for the small phosphoric oxide tube. On the outside of the reaction tube three lines are drawn (with ferric chloride solution) at distances of 3.75 cm, 7 cm and 7.5 cm from the square shoulder where the capillary joins the main tube, (*see* Note). The other end of the reaction tube is a silica capillary 7 to 8 mm external diameter and 1.5 mm bore, joined to the larger tube with a square shoulder.

NOTE — The mark at 3.75 cm indicates the position of the tip of the thermocouple.

B-1.1.2.7 *Thermocouples*

Made of platinum-rhodium and platinum, and provided with suitable millivolt meter for computing temperature. The thermocouple group is built round the silica thermocouple sheath about 5 mm in external diameter.

B-1.1.2.8 *Nitrometer*

Connected to the silica capillary through a three-way tap for collecting and measuring the volume of carbon monoxide formed.

B-1.1.2.9 *Kipp's apparatus*

For producing carbon dioxide.

B-1.1.3 *Reagents*

B-1.1.3.1 *Quality of reagents*

Unless specified otherwise, pure chemicals and distilled water [*see* IS 1070 : 1992 'Reagent grade water *(third revision*)] shall be used in the test (*see* Note).

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-1.1.3.2 *Reagents required*

a) *Nitrogen gas* — Cylinder nitrogen, purified by washing with alkaline pyrogallol solution and dried with concentrated sulphuric acid before entering and after passing through the preheater furnace.

b) *Alkaline pyrogallol solution* — Prepared by mixing 30 percent pyrogallic acid (*m/v*) solution and 60 percent (m/v) solution of potassium hydroxide in the proportion 1: 3.5.

c) *Concentrated sulphuric acid* [*see* IS 266 : 1993 'Sulphuric acid — Specification (*third revision*)'].

d) *Calcium chloride (anhydrous)* [*see* IS 1314 : 1984 'Specification for calcium chloride (*second revision*)']

e) *Phosphoric oxide*

f) *Carbon dioxide* — Prepared in Kipp's apparatus using hydrochloric acid and selected marble chippings (previously boiled with water for several hours). The gas is washed with sodium carbonate solution and dried by passing through concentrated sulphuric acid.

g) *Sodium carbonate solution* — 10 percent (*m/v*) solution.

h) *Concentrated magnesium chloride solution* — Saturated at room temperature

j) *Potassium hydroxide solution* — 40 percent (*m/v*) for use in the nitrometer.

B-1.1.4 *Procedure*

Insert a small pad of asbestos into the reaction tube and fill it with the prepared sample of coke up to the 7 cm mark, tapping the end of the tube lightly to settle the grains of the sample until no more can be added. Insert the thermocouple sheath into the reaction tube through the rubber bung. The level of sample in the tube rises to 7.5 cm mark due to the displacement of coke by the sheath and shows the apparent volume of coke when the thermocouple is in position. Then set up the apparatus.

Raise the temperature of the reaction tube (containing coke sample) from room temperature to 950 ± 2□ in a current of nitrogen duly purified by washing with alkaline pyrogallo solution and drying with concentrated

sulphuric acid, and preheated to 600 ± 2°C, about 1 000 ml of nitrogen and 1 h being taken for this. Maintain the temperature of the reaction tube at 950 ± 2°C for another 1 h and continue to pass nitrogen at the same rate. Stop nitrogen flow and then pass 100 ml of carbon dioxide [*see* **B.1.1.3.2 (f)**] prepared in the Kipp's apparatus at the standard rate of flow [*see* **B-1.1.1 (b)**] before starting the determination, start passing another 100 ml of carbon dioxide and collect the issuing gas in the nitrometer (**B-1.1.2.8**) over potassium hydroxide solution.

B-1.1.5 *Calculation*

$$\text{Reactivity of coke} = \frac{V_1}{V_2}$$

Where,

V_1 = Volume in millilitres, at NTP of carbon monoxide in the nitrometer as estimated by means of gas analyser; and

V_2 = Volume in millilitres, at NTP of carbon dioxide passed through the reaction tube.

B-1.2 Method B

B-1.2.0 *Outline of the Method*

The sampled and sized coke pieces are charged into a reaction tube and heated to 1 100°C in a stream of nitrogen/argon. Carbon dioxide is then passed at the rate of 51 per min for 120 min. The loss of mass after reaction is a measure of the rate of reaction of coke with carbon dioxide represented by $C + CO_2 = 2\ CO$. After reaction the coke loses its strength and to assess the loss in strength the reacted pieces are charged into an I-type drum which is rotated at a specified speed. The coke pieces are screened and the + 10 mm fraction taken as a measure of the strength after reaction.

B-1.2.1 *Standard Conditions*

The test conditions shall be as follows:

a)	Size of coke	19 to 21 mm
b)	Sample mass	200 g
c)	Test temperature	1 100˚C ± 5˚C
d)	Max rate of flow of nitrogen/ argon during heating and cooling	51 per min
e)	Heating time	45 min (*Max*)
f)	Soaking time at 1100˚C	15 min
g)	Rate of flow of carbon dioxide	5l per min
h)	Reaction time with carbon dioxide	120 min
j)	Revolution of I-Drum	30 min at 20 rpm
k)	Screening after rotation	10 mm (round hole)
m)	Dimension of I-Type drum after tester	130 mm dia × 700 mm tester

B-1.2.2 *Reactivity Apparatus*

The apparatus is illustrated in Fig. 2. The details of the essential components are as follows:

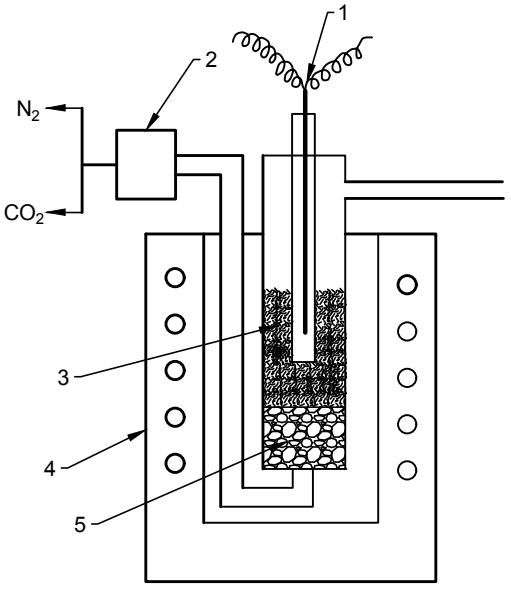


FIG. 2 APPARATUS FOR MEASURING COKE REACTIVITY INDEX

B-1.2.2.1 *Furnace*

Electrical resistance furnace maintainable at around 1 150°C, so as to obtain 1100 ± 5°C in the reaction tube shall be used.

B-1.2.2.2 *Reaction tube*

The tube shall preferably be made of good heat resistance material like Inconel-600. The internal diameter should be 76 mm. A 50 mm deep layer of refractory (such as fire clay) balls, 10 mm in diameter, shall be provided for proper distribution of gases. A thermocouple shall be inserted to the centre of the coke bed. The inlet and outlet pipes shall also be made of similar heat resistant material.

B-1.2.2.3 *Gas quality*

The inert gases (nitrogen/argon) shall be free of oxygen, moisture, carbon dioxide. A calcium chloride moisture trap shall be used. The reaction gas, that is, carbon dioxide shall be free from oxygen and moisture. The purity shall be 99 percent *Min.* A similar moisture trap shall be used.

B-1.2.2.4 *Flow meter*

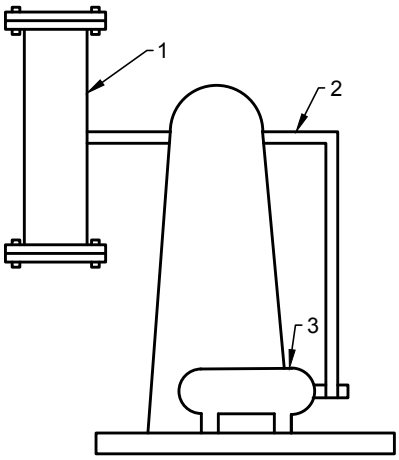
A standard rotameter or any other standard flow measuring device with proper control valve shall be included in the circuit.

B-1.2.2.5 *Safety measurement*

The exhaust gas from the reaction tube contains carbon monoxide and it should be either burnt by a pilot flame or disposed of at a suitable location away from the laboratory.

B-1.2.2.6 *I-Type drum tester*

A sketch is shown in Fig. 3. Necessary dimensions are given in **B-1.2.1.**



1 DRUM
2 BELT
3 MOTOR

FIG. 3 I-TYPE DRUM TESTER FOR MEASURING COKE STRENGTH

B-1.2.3 *Procedure*

The sampled coke pieces are dried thoroughly to constant weight at 105°C and charged into the reaction tube. The thermocouple is inserted at the proper location that is, middle of the coke bed. The reaction tube is placed in the furnace with nitrogen/argon switched on to prevent oxidation of coke pieces during heating. A soaking time of 15 min is allowed after the specified reaction temperature of 1100°C is reached. Nitrogen flow is stopped and carbon dioxide is then passed at 5 l/min for 120 min maintaining the temperature at 1 100 ± 5°C. After this carbon dioxide is stopped, furnace is switched off and cooling to room temperature is done in a stream of nitrogen/argon. Coke pieces are carefully withdrawn and weighed. The coke is transferred to the drum and rotated for 30 min at 20 rpm. The sample is then taken out and screened on a 10 mm round hole sieve.

B-1.2.4 *Calculation*

$$\text{Coke Reactivity Index (CRL)} = \frac{200 - w_1}{200} \times 100$$

$$\text{Coke Strength after Reaction (CSR)} = \frac{w_2}{w_1} \times 100$$

Where,

W_1 = Mass of coke in g, after reaction; and

W_2 = Mass of +10 mm in g, coke after reaction in I-type drum.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Solid Mineral Fuels and Solid Biofuels Sectional Committee PCD 07

Organization	*Representative(s)*
CSIR Central Institute for Mining and Fuel Research (CIMFR), Dhanbad	Dr Pradeep Kumar Singh (***Chairman***) Shri T. B. Das (*Principal*)
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Scientist 'C' (PCD), BIS

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BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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